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CRDEC-TR-305

SURVEY OF BIODEGRADATION OF ELECTRONIC COMPONENTS AND ASSOCIATED TESTING USING DECONTAMINATION SOLUTION

Darrel E. Menking
Irving Barditch
Paul Grasso

RESEARCH DIRECTORATE

August 1991

U.S. ARMY
ARMAMENT
MUNITIONS
CHEMICAL COMMAND

Aberdeen Proving Ground, Maryland 21010-5423

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
1991 August

3. REPORT TYPE AND DATES COVERED
Final, 89 Oct - 90 Sep

4. TITLE AND SUBTITLE
Survey of Biodegradation of Electronic Components and Associated Testing Using Decontamination Solution

5. FUNDING NUMBERS
PR-1C162622A553

6. AUTHOR(S)
Menking, Darrel E.; Barditch, Irving; and Grasso, Paul

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
CDR, CRDEC, ATTN: SMCCR-RSB, APG, MD 21010-5423

8. PERFORMING ORGANIZATION REPORT NUMBER
CRDEC-TR-305

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Biodegradation of electronic components was investigated from historical and experimental standpoints. A literature review was conducted on new materials. Twisted wire pairs were tested for degradation by decontamination solution (DS-2). Capacitance readings were used to determine degradation. Initial readings of the immersed wires dropped to zero, indicating shorting of the wire, followed by reading oscillation and return to approximately the initial reading. This phenomenon may be due to pinhole sealing. Further testing is necessary but will require the fabrication of a bridge capable of meeting ASTM/MIL STD specifications.

14. SUBJECT TERMS
Electronics — Microbes — Decontamination
Biodegradation — Fungi

15. NUMBER OF PAGES
26

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

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PREFACE

The work described in this report was authorized under Project No. 1C162622A553, CB Defense/General Investigation. This work was started in October 1989 and completed in September 1990.

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SURVEY OF BIODEGRADATION OF ELECTRONIC COMPONENTS AND ASSOCIATED TESTING USING DECONTAMINATION SOLUTION

1. INTRODUCTION

As part of the nuclear, biological and chemical (NBC) survivability technical base program development of test methods to assess the survivability of military hardware, a preliminary literature search of the biological effects on material was conducted. The results of this survey, and preliminary test results employing techniques used in earlier material effects studies, are reported.

The literature survey used all available databases from the Defense Technical Information Center (DTIC) in the "unclassified" category, open literature, and Dialog. The interaction of fungi and microbes with materials used in electrical/electronic equipment was a primary objective of the study; however, other materials such as fuels, structural materials and clothing were also noted (Appendix).

2. RESULTS OF SURVEY

During and after World War II, the impact of biota on materiel in tropical regions made the U.S. Army aware of biodegradation problems, especially with electronic equipment and water contaminated fuels. Electronic equipment at that time had a semiconformal coating of moisture and fungus proofing (MFP) varnish or lacquer, which contained mercury compounds; however, degradation remained a serious problem. To evaluate this problem in the electrical/electronic area, a comprehensive study was conducted for the U.S. Army Biological Laboratories (Fort Detrick, Frederick, MD) by Battelle Memorial Institute in 1964-1965.[1-3] This study covered all the materials such as epoxies, vinyls, and urethanes used in electrical/electronic systems as capacitors, circuit boards, insulators, resistors, etc. Organisms that caused the materials to degrade were obtained from infested electrical equipment from America and deteriorated equipment from the Panama Canal Zone. The fungi were evaluated by culturing growth on electrical components and conducting electrical tests to determine materiel degradation. Organisms of _Penicillium spp._ were among the most aggressive biota and, in some cases, penetrated the bodies of capacitors. The residue of the dead fungal hyphae then supported further microbial growth.

The first evaluation performed tested electronic components for fungal degradation using _Aspergillus_, _Penicillium_, _Alternaria_, _Streptomyces_, and _Rhodotorula_. Electrical parameter measurements, consisting of direct current (DC) resistance and dissipation factor, were made 160 days after inoculation with fungi. Detailed analysis of dissipation factor data showed that none of the capacitance measurements was outside the initial tolerance values. The Mann-Whitney U Test for two independent samples was applied to the resistance data. Results showed that although sensitive circuits would be affected, the majority of circuits would show no effects.

The second Battelle evaluation performed tested for "bridging" of fungi across insulating materials. For this evaluation, printed circuit board (PCB) test specimens were prepared and assayed for fungal bridging to test for electrical leakage, and peel strength measurements were taken to test for material degradation. These data were also subjected to the Mann-Whitney U Test that was used in the first trial. Results indicated that the bridging would impair the performance of high impedance and critically tuned circuitry. The coaxial cable showed serious degradation, which could impair the performance of electronic items containing these parts. Peel test measurements showed no degradation of PCB materials. Fungi of the genera _Aspergillus_, _Alternaria_, _Sphaeropsis_, and _Penicillium_ were used in this study.

The Battelle report was very comprehensive; however, materials introduced in the past 25 years have not been subjected to an equally thorough evaluation. In addition, earlier studies failed to test impedance over an appropriate frequency range, relying solely on DC resistance measurements to detect electronic failure conditions. Some of the materials recommended for future study were silicone, polyamide, teflon, epoxy, polyurethane, vinyl, neoprene and bakelite.

3. EXPERIMENTAL PROCEDURES

3.1 Discussion.

In the Battelle report, material testing was done with DC only; however, DC testing alone does not evaluate all the pertinent performance parameters involved in equipment. Testing using alternating current (AC) measures both resistivity and capacity (AC impedance), which should be assessed because changes in the parameters show that the insulator's dielectric properties are being degraded. Capacity failure of the conformal coating protecting a wire/circuit would render the AC transmission nonfunctional even though the wiring still retained a fairly high DC resistivity. To test the practicality of using AC impedance methods to assess the material effects of biological spores and organism by-product challenges, we conducted a series of capacitance/resistance measurements of conformal coated wire. As part of this effort, test methods are being developed to assess the effects of biological degradation on the performance of electrical insulation and protective coatings for circuit boards and wires. Fortunately, these parameters can be measured easily in a nondestructive, _in situ_, real time manner by an AC bridge of the requisite sensitivity. Materials should also be tested at different frequencies because the material components respond to different frequencies. For example, ionic components respond to DC, and dipole components respond to AC. In the latter case, when resonant frequency is reached, the wire overheats and is no longer useful as a transmission line.

Conformal coatings (polymeric materials) were selected because they have many applications, including serving as insulators in electrical or electronic systems. These coatings' behavior following exposure to chemicals (agents/decontamination solutions) associated with chemical warfare are of great interest to the military in battlefield applications. To alter the electrical characteristics of the test coatings, the wires were exposed to decontamination solution (DS-2). Though it could not be considered a biological simulant, DS-2 did provide us with useful material data. An AC impedance measurement technique using a twisted wire pair,[4] with and without external bias, was conducted at frequencies of 120 Hz and 1 kHz to determine the suitability of using this method to assess the interactive effects of the chemical, coating, and voltage.

3.2 Description.

Pieces of vinyl-insulated #20 wire twisted at 10 turns/inch and having a surge impedance of 120 ohms were placed on the measuring terminals of a General Radio (GR) 1639 Digibridge for testing without external bias. After recording the pretest value of capacity and resistance, the wire pair was immersed in DS-2, and the change in capacity and resistivity were tracked. Two samples were tested, one having been irradiated to increase polyvinyl crosslinking.

Samples of wire were also tested with and without an external bias of 27 V to test for additional breakdown due to the bias. The dielectric constant at increased voltage may become nonlinear, at which point resistivity decreases, and energy losses increase due to material overheating. Unlike the preliminary test wires, these were monitored past the breakdown point.

4. RESULTS

4.1 Preliminary Testing.

For both wires, the capacity increased until failure occurred, an event marked by a sudden drop to zero. During this period, the resistivity dropped by a factor of 4. The capacity for the untreated sample failed after 9 min (Figures 1 and 2); the irradiated sample failed after 20 min (Figures 3 and 4). The wires were washed with deionized water and remeasured at somewhat lower capacity levels due to the chemical permeation of the wires.

4.2 Testing Without External Bias.

Another test was carried out on a sample of Belden red vinyl-covered hookup wire to determine the effects of external bias. In two test runs, the wires were prepared as a twisted pair, and part of the length was immersed in DS-2. The values of capacity and loss factor (D) were measured in air and distilled water. The wire pair was then placed in DS-2. After about 10 min (in both tests), the capacity reading intermittently dropped to zero indicating a short between the wires. No value for the associated resistance part of the short could be measured, indicating the values exceeded 10^9 ohms, the upper measurement range for the GR Digibridge.

Upon continued immersion, the wire appeared to revert to approximately its original state. After several days, the resistance value between the wire

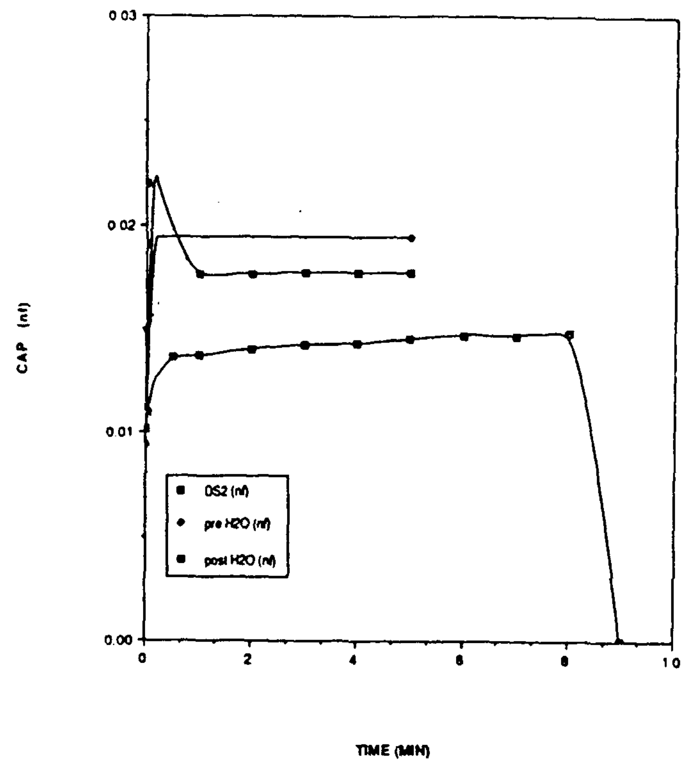

Figure 1. Twisted Wire Test-DS2 (Red/PVC/20 Gage) Capacitance

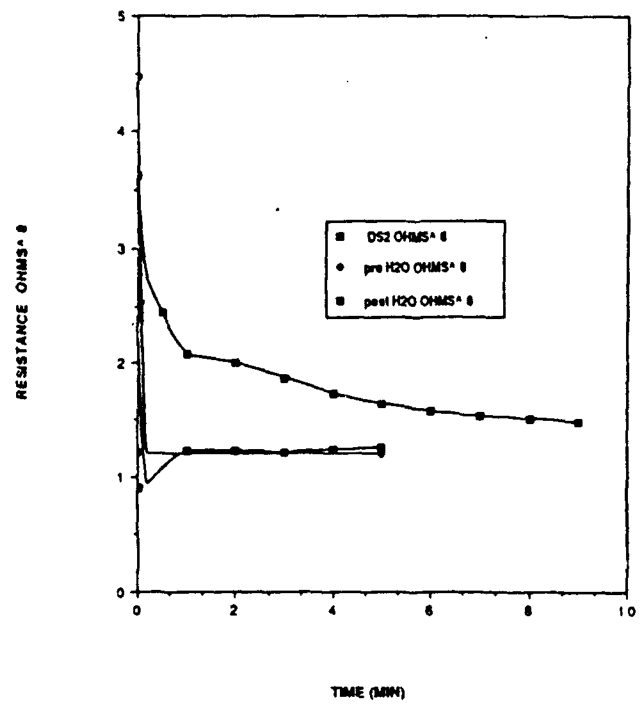

Figure 2. Twisted Wire Test-DS2 (Red/PVC/20 Gage) Resistance

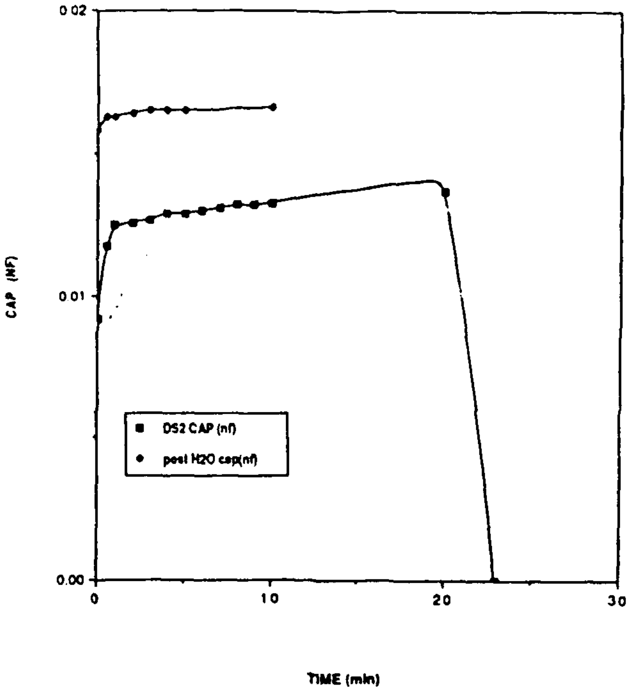

Figure 3. Twisted Wire Test-DS2 (Yellow/PVC Irad/24 Gage) Capacitance

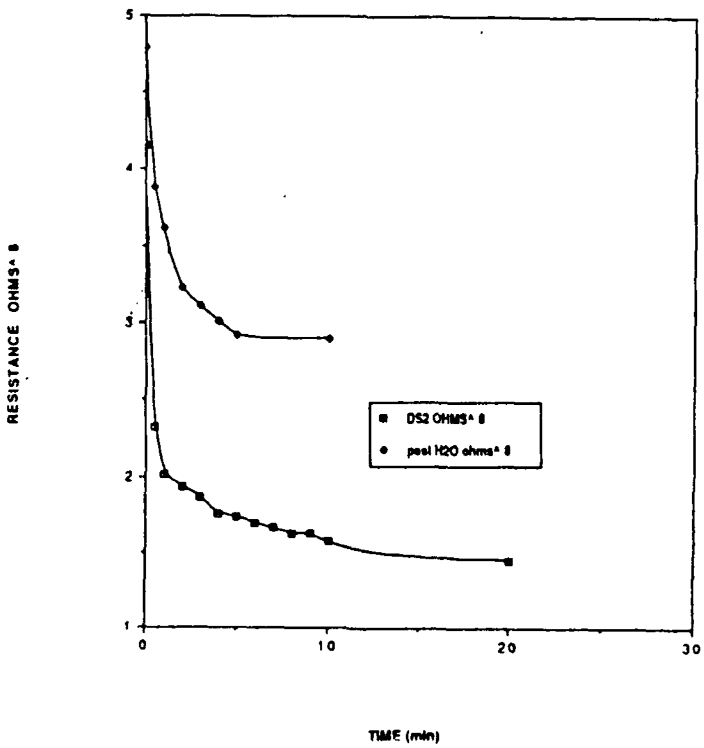

Figure 4. Twisted Wire Test-DS2 (Yellow/PVC Irad/24 Gage) Resistance

again dropped below 108 ohms (Figures 5 and 6). The pigment in the red vinyl wire became black, indicating a chemical reaction; however, additional examination of the vinyl insulation is needed to determine precisely what processes occurred during the exposure period. One implication of the measurements is that there is an inductive component present in the equivalent circuit for the insulation. Measurements made at 120 Hz and 1 kHz differed, indicating a frequency sensitivity in the system unlike that which would be due to capacity alone. Additional work, such as electron microscopy and materiel stress testing, is recommended to establish a better understanding of this phenomenon.

4.3 <u>Testing with External Bias</u>.

Testing with external bias showed no significant changes from testing without bias. The time to failure was approximately 9-20 min, followed by a return to the original state, for the two wire types, regardless of applied bias. In all cases (Figures 5 and 6), the failure point was identified as a capacitance short.

5. DISCUSSION

5.1 <u>Discussion of Results</u>.

The AC impedance is a very sensitive and practical test for chemical and biological deterioration of materiel. A small external bias has no effect on the rate of deterioration and, therefore, would not need to be pursued for many low voltage applications. There may be a need for high voltage testing -- 500 V. The failure process can be very complex, so a full spectra of frequency is important.

In each case, the capacitance increased, and the resistance decreased with time. Dielectric changes were greatest during the initial 30 sec. This phenomenon was probably due to the wetting process, as the wires went from an air to a DS-2 insulation system. About 10 min after these wetting changes occurred, there was a period in which capacitance oscillated between zero and the initial readings. This event lasted for about 2 min, after which readings returned to the expected nonzero values. It is hypothesized that micropores formed and filled with DS-2, which shorted the circuit until the surrounding polymer was able to swell and seal the holes. Surface analysis of this phenomenon has begun to confirm this theory. After a few minutes, all wetted pores were sealed, and the slower diffusion of the DS-2 into polyvinyl chloride (PVC) continued to deteriorate the coating's dielectric properties in a monotonic fashion.

This pinhole process may be similar to the formation of an oxide layer in aluminum electrolytic capacitors immersed in a borax solution. In this system, a film of aluminum oxide forms as current passes through the electrodes. The inorganic oxide layer forms a capacitive element, which breaks down through the pinholes of nonoxidized aluminum. As time progresses, the current follows the best conductive path through the pinholes, which in

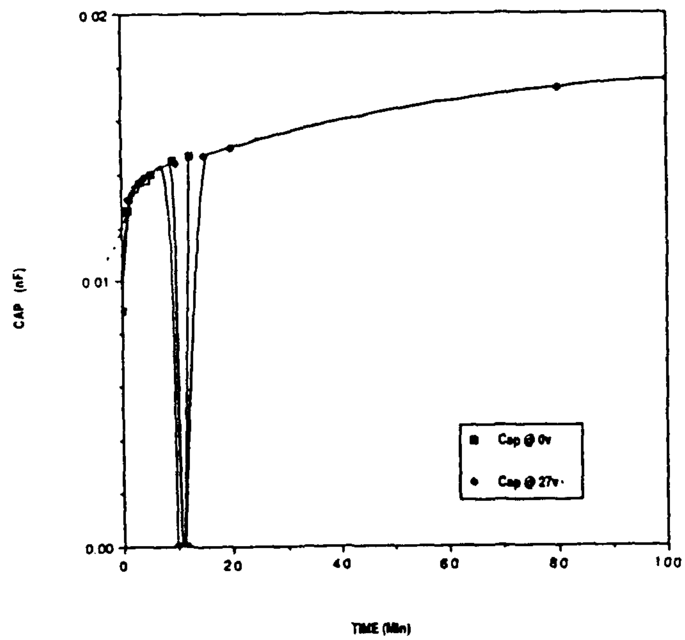

Figure 5. Twisted Wire Test-DS2 (Red/PVC/20 Gage/0 and 27 V Bias/1 kHz) Capacitance

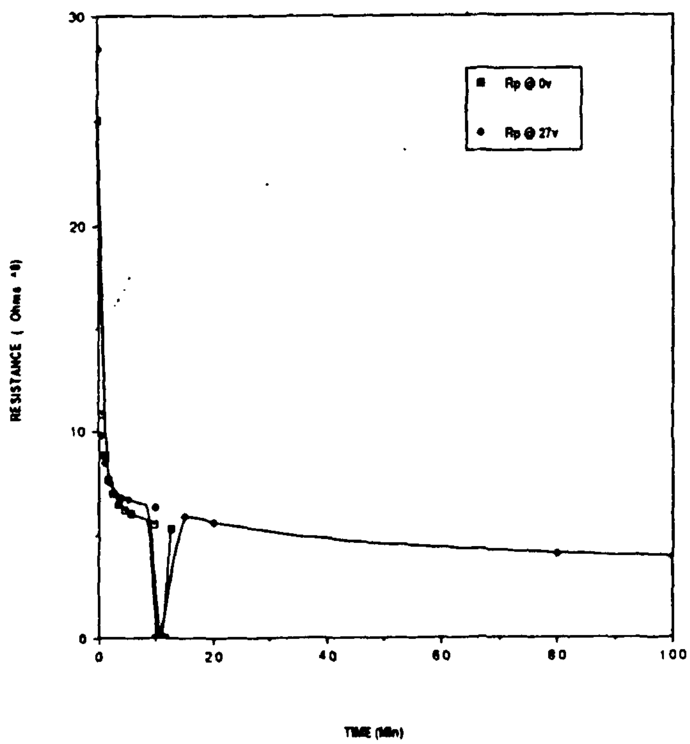

Figure 6. Twisted Wire Test-DS2 (Red/PVC/20 Gage/0 and 27 V Bias/1 kHz) Resistance

turn seals the conductive sites. The vector for this process is an electrical current interacting with a metal coating, whereas, in our case. the DS-2 interacts with a PVC coating.

Temperature change did not affect the long term capacitance readings (Figure 7). However, resistance increased when sunlight came in contact with the solution, but dropped when there was no solar heating. For this reason. days 3 and 4 were dropped from the linear fits in the long term effects (Figure 8). Future studies need to be environmentally controlled to minimize the effects of temperature and relative humidity (RH) on experimental results.

The outer surface of the wire blackened within about 2 min of its exposure to DS-2. The involvement level was limited to the upper layers of the coating and could not be rubbed off. At this point, we do not know if this was an attack on the red pigment or an insulation failure; however, the bulk insulation behaves differently than the surface layer. This could be the result of curing differences, the method of applying the insulation to the wire, or applied strain introduced during the twisting process.

5.2 Need for Further Testing.

Because many new materials, which are coming into wider military use in electronic equipment and microprocessors, have been introduced in the past 25 years, a comprehensive evaluation should be conducted. Some examples of new insulating material that need to be tested are parylene, kaptanes and fluorinated synthetics.

An evaluation of the survivability of new materials would consist of identifying the biota that would likely attack the materials, culturing those organisms, and testing the degradation under laboratory-controlled conditions. The conditions required for organism growth and testing may require a P3 facility because organisms of unknown pathogenicity may be used for testing, and this type of testing could involve significant expense.

5.3 AC Bridge Design.

Several commercially available bridges, such as the DuPont DSA-2970, Hewlett-Packard 4194A, and the Spatial Dynamics Applications M-24 Microwave Dielectrometer, have the capabilities specified in the applicable American Society for Testing and Materials (ASTM) and Military Standards (MIL STD) specifications; however, no one machine covers all the frequency bands of interest. Therefore, it is necessary to design and fabricate a bridge with unique features to meet these standards. One requirement is that testing be performed at high frequencies of 10 and 100 MHz. At these frequencies, resistors of high values (10^{12} ohms) are overshadowed by stray capacitance readings. Calculations show that the values are expressed as a capacitor parallel with a resistor (Table 1). The relationship between the standard resistor (S) and the test resistor (R) has a scale factor of $1/(wc)^2$. Table 2 lists sample calculations of value ranges.

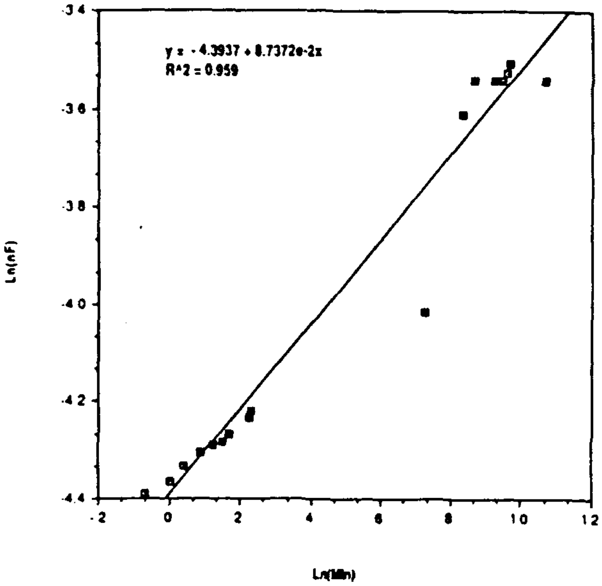

Figure 7. Long Term Capacitance Effects (0 V Bias)

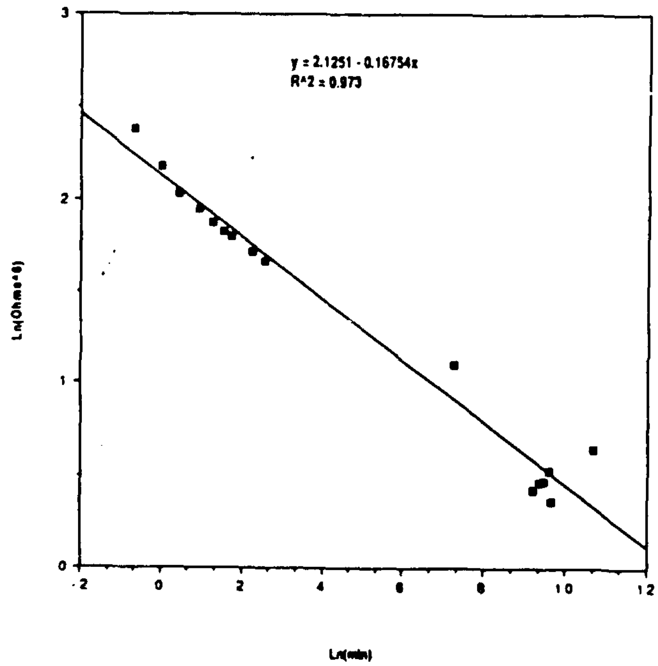

Figure 8. Long Term Resistance Effects (0 V Bias)

Table 1. Series-Parallel Transformation

$$R_s + \frac{1}{(j\omega C_s)} = \frac{R_t\ (1/\ j\omega C_p)}{R_t + (1/\ j\omega C_p)}\ \cdot\ \frac{(j\omega C_p)}{(j\omega C_p)}$$

$$R_s + (\frac{1}{(j\omega C_s)})(j/j) = \frac{R_t}{(1 + j\omega R_t C_p)}\ \frac{(1 - j\omega R_t C_p)}{(1 - j\omega R_t C_p)}$$

$$R_s - j(1/\omega C_s) = \frac{R_t}{1 + (\omega R_t C_p)^2} - j(1/\omega C_p)$$

Hence:

$$R_s = \frac{R_t}{1 + (\omega R_t C_p)^2}$$

And:

$$C_s = C_p$$

Where:

R_s = SERIES RESISTOR
R_t = TEST RESISTOR
C_s = CAPACITOR IN SERIES
C_p = CAPACITOR IN PARALLEL

Table 2. Sample Calculations for Transformation

The following resistance values are calculated at 100 MHz and capacitance of 10^{-12} f.

$$S = 1/10^8 \times 10^{-12})^2\ R$$

S	R
10^2	10^6
10	10^7
10^0	10^8
10^{-1}	10^9
10^{-2}	10^{10}
10^{-4}	10^{12}

These calculations indicate that to express the test subject wires as series resistance (R_s) calls for unreasonably low values of resistance. Series resistance values of less than 10^{-1} ohms are small enough to cause serious difficulty in use. The insulation resistance of parylene is about 10^{12} ohms. Lesser test values can be measured if they are from 10^6 to 10^9 ohms. As a compromise, standard sets of insulators of known values may be used in the standard arm to make the comparison with a degree of accuracy. The standard resistor set can be made of ceramic set to a given bulk resistivity. Used with button connectors, a set of resistance values may be achieved, and a variable capacitor whose rotor is grounded may be used to set a balance. An alternative system uses twisted wire pairs in all bridge arms; null detection and radio frequency (RF) drive remain the same. The second feature is the need to make this measurement at operational values of about 100 V or higher.

Among the types considered, the Scheering type bridge is the most suitable (Figure 9). This bridge comprises two capacitor arms (standard arm and a test arm). The balance detector system will comprise a back diode, ferrite decoupling, and shielding with an instrumentation amplifier to sense a DC null. The various frequencies for test and the bridge drive signal can be derived from fixed frequency oscillators at 10 and 100 MHz, thereby eliminating the need for a variable frequency source. Tuning and transformers for the RF will be needed to drive the bridge. A linear amplifier of suitable power rating will be fed from the crystal oscillator with suitable impedance matching devices. Successful operation of this system will allow testing in full conformity with the applicable ASTM specifications.

6. CONCLUSIONS

Testing new dielectric materials should be done for chemical and biological degradation in accordance with ASTM and MIL STD specifications, which include all the pertinent parameters. Minimum modification of the preliminary testing procedures will make it suitable for evaluating degradation due to the various biota. Because no commercial bridge has the capabilities specified in the applicable ASTM/MIL STD specifications, a bridge with unique features to meet these standards must be designed and fabricated.

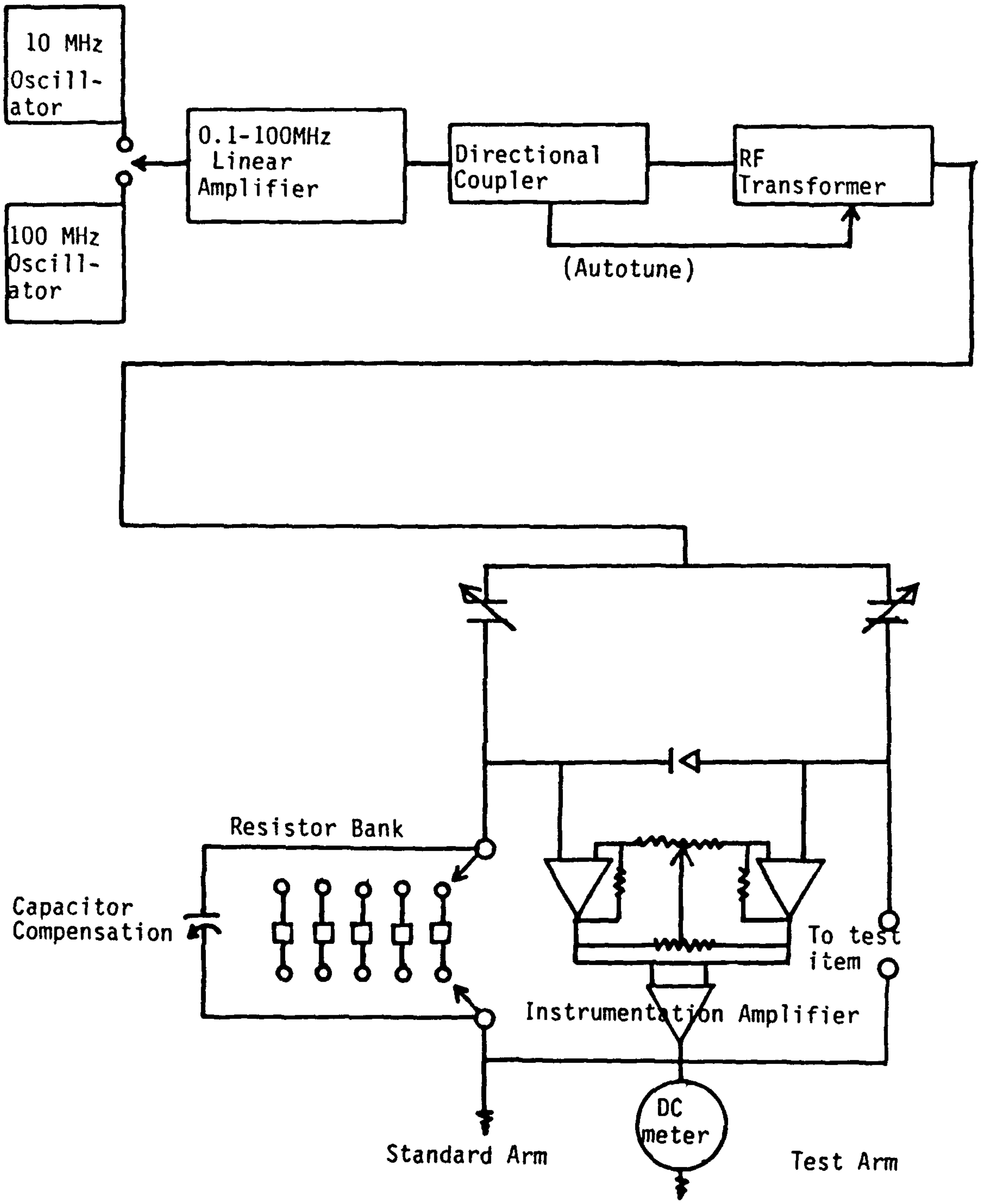

Figure 9. Scheering Bridge and Ancillary Equipment

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